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(54) Hard layer coated parts

(57) In hard layer coated parts which are coated with hard layers, this invention aims at improvements of wear resistance, oxidation proof and lubrication properties.

In hard layer coated parts which are coated with hard layers, hard layer coated parts featured by hard layers coated with minimum 1-2 layers which contain Al, Ti, Cr, N, O.

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Description

[0001] This invention is about wear-resistant parts with higher solidbody lubrication capability as well as higher wear resistance and oxidization proof.

[0002] In the field of cutting tools, moulds and mechanical components, it is popular to coat various hard layers in order to have wear resistance, oxidation proof, lubrication capability. Typical TiN, TiCN layers have good wear resistance, but still have problem of oxidation proof. Furthermore, TiAlN based layer proposed by Japanese laid-open patent specification Sho62-56565 and others have good wear resistance and oxidation proof but lubrication capability is still low. CrN, CrCN based layers have good lubrication capability, but have lower layer hardness and lower wear resistance. Like the above, conventional layers have inferiority in either wear resistance or oxidization proof or lubrication property and still have some problems in various applications. In addition, in order to have lubrication property, Japanese laid-open patent Hei 5-239618 and others proposed to coat MoS based layer which has better lubrication property on the surface of hard layers, however adhesion is poor and does not have enough results. Like this, conventional layers still have a certain problem and in order to solve problems with layers other than MoS based layer, Japanese laid-open patent Hei 11-156992 proposed to coat CrN based layer on the top layer of TiAlN based layer, but not yet satisfactory in wear resistance, because thickness of TiAlN layer is not enough, due to limitation of the entire layer thickness, to some extent.

[0003] The purpose of this invention is to improve wear resistance, oxidation proof and lubrication property without degrading any one of all those properties.

[0004] In order to solve above mentioned themes, in this invention, in hard layer coated parts which is coated with hard layers, hard layers are coated with minimum one or two layers which contain Al, Ti, Cr, N, O. Furthermore, superior execution modes of this invention are:

- Chemical analysis of each layer which consists hard layer is:

$$(Al_aTi_bCr_c)\,(N_wO_{1-w}),$$

however, $30 \leqq a \leqq 70$, $30 \leqq b \leqq 70$, $0.5 \leqq c \leqq 20$,
$a+b+c=100$, $0.7 \leqq w \leqq 0.99$,

- The number of layers is 3-1000 layers.
- Thickness of each layer is 5 nm-2000 nm.
- Hard layer consists of less oxide-containing A-layer and more oxide-containing B-layer.
- Oxigen content of A-layer is 1-10 atomic %, while oxigen content of B-layer is 10-30 atomic %.
- In partial or entire layers, oxigen content is inclination composition.
- Crystal structure of hard layers is NaCl type.
- In X-ray diffraction of hard layer, supposing that the intensity of the diffraction of (200) plane is 1(200) and the intensity of the diffraction of (111) plane is I(200), I(200)/I(111) = more than 1.
- Crystal structure of hard layers is fine columnar crystal or amorphous.
- Grain diameter of fine columnar crystal is smaller than 250 nm at a distance of 1000-1500 nm from the border-plane hard layer and substrate.
- Compression stress residual in hard layers is less than 3,5 GPa.

[0005] This invention is adoption of hard layers to which oxigen is added, while Ti, Al, Cr and N are essential elements. Naturally, Ti and Al contribute as wear resistant elements and Cr contributes as element which gives lubrication property, however, these are not sufficient and therefore by adding oxigen, stronger oxidization proof and lubrication property are gained.

[0006] In the field of cutting tools, first of all, oxidation proof is further improved, when Cr is added to TiAlN-substrate. In case of TiAlN, it is well known that along with oxidation, inside the layer Al is diffused to the surface and by creation of Al oxide, oxigen penetration from outside is supressed resulted in an improvement of oxidation proof. However, in this case, when expecially a shock of cutting tool is given, Al oxide can easily plicks-off and it is difficult to keep that effect, because underneath Al oxide, very porous Ti oxide is created. The same goes to moulds and the like. It was proved that porous Ti oxide created underneath Al oxide turns into TiCr oxide by adding Cr and this oxide forms very dense layers. Accordingly, Al oxide created on the top layer has sufficient adhesion and in result, oxidation proof is improved.

[0007] The second effect of Cr addition is, god lubrication property Cr itself is given to TiAlN layer. Friction co. efficiency of TiAlN against steel is 0,7 - 0,8, but along with Cr addition, it can be improved to 0,3 - 0,6. This friction co. efficiency depends on the volume of Cr added. However, when the volume of Cr addition is too much, it causes decrease

of layer hardness resulted in inferior wear resistance and therefore it is better to settle upper limit of the volume of addition.

[0008] It is confirmed that Cr addition only can improve lubrication property and oxidation proof of TiAlN based layers, but Cr is not enough and further improvement is recognized when oxigen is added. Effect of oxigen addition results, first of all, drastic improvement of oxidation proof as well as drastic improvement of lubrication property. It is considered, the reason why oxidation proof is drastically improved is that along with oxigen addition inside the layer, crystal becomes finer and layer itself becomes dense and grain boundry becomes dense so that the speed of oxigen diffusion against oxigen penetration from outside is drastically supressed. Improvement of lubrication property has not yet been analyzed well but it is considered, its reason is that affinity with steel becomes lower by adding oxigen.

[0009] The second effect of oxigen addition is that wear resistance is improved by improved adhesion of layers, due to lowering of residual compression stress in layers. Adhesion of layers is critically important especially in heavy duty cutting or in the field of forging dies. There is a trend of wear progress caused by small peeling-off of layers and when big peeling-off takes place, life times comes to an immediate stop. Peeling border load in scratch test of AlCrN based layer is 60-80N, while it is improved to more than 100N by adding oxigen.

[0010] However, when the volume of oxigen addition increase, wear resistance improves, because of above mentioned improvements of oxidation proof, lubrication property and adhesion, but on the other hand, layer hardness itself is softened resulted in inferior abrasive wear resistance. Accordingly, it is important and desirable to make multi-layers of layers with optimized elements which contribute to oxidation proof and lubrication property and layers with optimized elements which contribute to abrasive wear resistance. Advantages of the above two kinds of layers are multiplied by making multiple layers.

[0011] In the next place, the reason why values were limited is explained. In case Al is less than 30 atomic %, oxidation proof of layers becomes worse, while it is more than 70 atomic %, AlN with hcp structure created in layers makes layer-strength weaker and therefore undesirable. In case Ti is less than 30 atomic %, wear resistance of layers becomes worse, while when it is more than 70 atomic %, oxidation proof of layers becomes worse and therefore undesirable. In case Cr is less than 0,5 atomic %, porous Ti oxide is created which does not contribute to improvement of oxidation proof, while it is more than 20 atomic %, layer hardness is softened and wear resistance becomes worse and therefore undesirable. In case oxigen is less than 1 atomic % against nitrogen, it does not contribute to improvement of oxidation proof, lubrication property and adhesion, while it is more than 30 atomic %, layer hardness is softened and therefore undesirable.

[0012] When the number of layers in multi-layers is less than three layers, though they show individual effects, as mentioned above, either defect becomes remarkable and multiplied effects cannot be observed. On the other hand, when the number of layers is more than 1000 layers, each layer thickness is too thin which does not bring multiplied effects and at the same time there is a trend of increase of residual stress and loses adhesion property of the layers and therefore undesirable. The same goes to each layer thickness. When each layer thickness is less than 5 nm, effects of advantages of each layer are weakened, while when it is more than 2000 nm, only approx. three layers are realized and therefore undesirable.

[0013] As mentioned above, the purpose of multi-layers of low oxigen-containing layers and high oxigen-containing layers is, low oxigen-layers have smaller hardness decrease and contribute to abrasive-wear resistance, which high oxigen containing layers greatly contribute to oxidation proof, lubrication property, though there is a trend of decrease of layer hardness. By coating these into multi-layers, both effects are multiplied and bring favourable efffects. In low oxigen containing layers, when oxigen containing volume is less than 1 atomic %, adhesion with high oxigen-containing layers is weakened, while it is more than 10 atomic %, abrasive wear resistance is degraded and therefore undesirable. On the other hand, in case of high oxigen containing layers, when oxigen containing volume is less than 10 atomic %, it does not contribute so much to improvement of oxidation proof, lubrication property, while it is more than 30 atomic %, layer hardness is drastically softened and loses wear resistance and therefore undesirable.

[0014] Simple multi-layers of these low oxigen containing layers and high oxigen-containing layers can create no problems, but adhesion of each layer is further improved either by inclining oxigen content in each layer and minimizing changes of oxigen contents at border-planes or by making oxigen contents continuous like sine curve.

[0015] In crystal structure, NaCl type has many sliding surfaces and layer hardness in high temperature has an upper limit of approx. HV3000 and it is difficult to have higher hardness. On the other hand, it has better ductility, smaller creation of chippings, smaller creation of micro cracks when a shock is given and therefore stable life time can be achieved.

[0016] Crystal orientation of layers depends on coating conditions. When there is a trend that when depositioning with relative low energy, it is strongly oriented to (200) plane, while when depositioning with relative high energy, it is oriented to (111) plane. It was confirmed that in case of deposition with low energy, deposition rate of layer is low, but layer density is improved and results in better oxidation proof and wear resistance. Accordingly, it can be said when (200) plane intensity of the diffraction is stronger than the one og (111) plane, more superior oxidation proof and wear resistance are gained and therefore more favourable. Crystal orientation does not affect lubrication property so much.

[0017] Crystal grain diameter of layer is decided at fractional surface SEM and draw a line parallel to base body at a distance of 1000 nm - 1500 nm from subtrate surface and prescribed by the number of grain boundary which cross the line. In this case, crystal grain diameter in the layer is bigger than 250 nm, wear resistance, layer strength degrade and therefore undesirable. State of amorphous means in this case that it is not amorphous actually, however clear crystal grain boundary cannot be observed in observation of fractional surface. In such a case especially, a remarkable improvement of oxidation proof is confirmed.

[0018] Compression stress residual in layer depends on coating conditions, but when exceeding 3,5 GPa, adhesion is degraded and therefore undesirable. By the way, the layers of this invention can have the same trend in production system of Arc Ion Plating, Sputtering, Electron beam-evaporation, Plasma Assist CVD and production method can be combination of those production methods.

[0019] In the next place, favourable embodiment in this invention is explained hereunder together with comparison examples. Sample layers of this invention and comparison samples were produced in Arc Ion Plating. Composition of AlTiCr was adjusted by adjustment of metal composition of cathod target which are evaporation source. Oxigen content was adjusted by mixing ratio of mixed gas of nitrigen and oxigen and also by switching over gasses. Crystal orientation is basically adjusted by coating conditions and (200) orientation layers were produced by coating conditions with 70 V bias voltage which is given to the substrate/reaction pressure 1 Pa, while (111) orientation layers were produced with 200 V bias voltage/reaction pressure 0,5 Pa. Besides, ratio I(200)/I(111) depends a little also on layer composition and oxigen containing volume.

Chart 1:

	test pc No,	A-layer	B-layer	No. of layers
examples of this invention	1	50Al40Ti10Cr-5095N	50Al40Ti10Cr-25075N	20
	2	55Al35Ti10Cr-5095N	55Al35Ti10Cr-25075N	20
	3	35Al55Ti10Cr-5095N	35Al55Ti10Cr-25075N	20
	4	65Al32Ti3Cr-5095N	65Al32Ti3Cr-25075N	20
	5	33Al64Ti3Cr-5095N	33Al64Ti3Cr-25075N	20
	6	40Al35Ti25Cr-5095N	40Al35Ti25Cr-25075N	20
	7	50Al40Ti10Cr-2098N	50Al40Ti10Cr-25075N	20
	8	50Al40Ti10Cr-5095N	50Al40Ti10Cr-13087N	20
	9	50Al40Ti10Cr-5095N	50Al40Ti10Cr-25075N	4
	10	〃	〃	100
	11	〃	〃	500
	12	〃	〃	900
	13	50Al40Ti10Cr- N inclination (10-1-10) 0 (90-99-90) N傾斜	50Al40Ti10Cr- N inclination (10-25-10) 0 (90-75-90) N傾斜	20
	14	40Al35Ti25Cr-5095N	65Al32Ti3Cr-15085N	20
	15	33Al64Ti3Cr-7093N	40Al35Ti25Cr-15085N	20
comparison examples	16	TiN	-	1
	17	Ti-50N50C	-	1
	18	50Al50TiN	-	1
	19	TiN (500nm)	50Al50TiN (2500nm)	2
	20	65Al35TiN	-	1
	21	65Al35TiN	100nmMoS$_2$	2
	22	65Al35TiN (2000nm)	CrN (1000nm)	2
	23	50Al50Ti-70N30C	-	1
	24	TiN	50Al50TiN	20
	25	50Al40Ti10CrN	-	1
	26	50Al40Ti10Cr-5095N	-	1
	27	50Al40Ti10Cr-25075N	-	1
	28	50Al40Ti10Cr-5095N	50Al40Ti10Cr-50050N	20
	29	〃	〃	1500

[0020] In Chart 1, examples of this invention and comparison examples are shown. Layer thickness of examples of this invention as well as in comparison examples are all 3000 nm - 3200 nm.

Chart 2:

	test pc No.	hardness (HV)	I (200) / I (111)	weight increase by oxidation mg/min	friction co.efficiency	stress Gpa	grain dia. nm
examples of this invention	1	2650	5.23	1.87	0.35	1.26	160
	2	2700	4.22	1.11	0.34	1.35	148
	3	2570	7.11	3.56	0.38	1.05	220
	4	2750	3.25	1.43	0.33	2.04	158
	5	2490	4.33	3.10	0.32	2.22	210
	6	2500	2.89	0.98	0.38	1.59	amorphous
	7	2670	2.56	1.98	0.39	2.46	201
	8	2710	2.77	2.31	0.40	2.41	175
	9	2630	6.14	1.78	0.35	1.02	210
	10	2690	4.26	1.55	0.34	1.96	143
	11	2750	3.44	1.43	0.33	2.31	121
	12	2800	2.49	1.40	0.33	2.93	98
	13	2660	5.03	1.79	0.32	1.76	158
	14	2600	4.34	1.23	0.38	1.88	125
	15	2590	3.89	1.49	0.33	1.97	128
comparison examples	16	2160	0.25	85.28	0.87	1.67	350
	17	2980	0.22	98.13	0.29	4.02	256
	18	2700	2.43	11.67	0.85	2.89	341
	19	2700	0.56	12.55	0.85	3.97	298
	20	2750	3.22	8.05	0.89	2.11	253
	21	2720	3.22	22.23	0.11	2.78	331
	22	2240	3.22	15.67	0.29	2.88	332
	23	3010	2.56	25.44	0.65	3.97	247
	24	2450	2.33	56.81	0.88	2.85	286
	25	2680	4.88	6.45	0.55	3.63	194
	26	2600	6.22	3.27	0.53	2.10	154
	27	1920	8.72	0.78	0.24	0.89	amorphous
	28	1930	6.32	0.98	0.28	1.11	23
	29	1980	6.08	0.76	0.27	1.56	amorphous

[0021] In Chart 2, measuring results of examples of this invention and comparison examples shown in Chart 1 are explained, concerning oxidation proof, lubrication property and wear resistance to which layer hardness contributes. For oxidation proof, weight increase/unit time by oxidation by holding test pcs at 900°C in open air was measured. Lubrication property was analyzed by measuring friction co.efficiency with carbon steel. For hardness, vickers hardness was measured by prove ball penetration depth under 1 g load, using nano indenter. It is very clear that examples of this invention are superior to comparison examples in every point.

Chart 3:

	test pc No.	end mill life (m)	drill: thrust/N	No. of holes	life of inserts hr
examples of this invention	1	65	125	760	1.54
	2	75	120	950	1.78
	3	48	127	578	1.22
	4	81	135	1016	1.88
	5	55	137	783	1.35
	6	55	116	852	1.41
	7	60	127	679	1.49
	8	51	132	653	1.45
	9	60	128	720	1.44
	10	69	120	823	1.60
	11	71	110	954	1.75
	12	75	108	1036	2.01
	13	87	121	979	1.86
	14	63	128	857	1.56
	15	61	129	891	1.60
comparison examples	16	2	195	21	0.11
	17	4	101	43	0.24
	18	27	189	257	0.75
	19	25	185	298	0.77
	20	31	186	358	0.81
	21	34	175	348	0.75
	22	29	115	211	0.45
	23	35	165	278	0.71
	24	14	190	86	0.33
	25	30	150	364	0.85
	26	36	140	484	1.03
	27	8	105	112	0.16
	28	12	101	153	0.31
	29	13	95	143	0.22

[0022] In Chart 3, tool life of examples of Chart 1 is shown through end mill cutting under conditions below.

Substrate composition	90WC - 9,5 Co - 0,5 Cr, WC grain dia. 0,8 µm
Tool	6 cutting blades, dia. 8 mm end mill
Cutting object	SKD 11 (HRC 63)
Cutting speed	100 m/min
Depth of cut	8 mm x 0,8 mm
Feed rate	50 µm/cutting edge
Dry or wet	Dry cutting

[0023] Criterion of tool life judgement is when end mill is broken into two pieces. In any respect, tool life of examples of this invention is longevity and effects of multi-layer structure with TiAIN base added by Cr and oxigen are self evident.

[0024] In Chart 3, results of hole-drilling of examples of this invention and comparison examples in Chart 1 with the conditions below are also described. Thrust power is the result of measurement at 10th hole at initial stage of drilling. Tool life was judged when drill was broken.

Substrate composition	91,5WC - 8 Co -0,5 Cr, WC grain dia. 0,8 μm
Drilling object	SKD 61 (HRC 42)
Drill dia.	8 mm
Cutting speed	80 m/min
Feed rate	0,2 mm/rev.
Depth of hole	32 mm
Dry or wet	Dry cutting

[0025] It is self evident that examples of this invention has remarkably low thrust resulted in longevity.
[0026] In the next place, hard metal inserts of this invention and comparison were put into cutting test. Its results are also described in Chart 3. In case of front milling, oxidation proof is important, because cutting speed is high.

Substrate	P30 grade hard metal alloy
Insert	SEE42TN (clearance angle is 5°)
Cutting object	SKD61 (HRC22)
Cutting speed	400 m/min
Cutting depth	1 mm
Feed rate	0,1 mm / cutting edge
Dry or wet	Dry cutting

[0027] Judgement of life was cutting time until average wear of relief face reached 0,4 mm.
[0028] As clear from Chart 3, remarkable improvement of tool life of examples of this invention was confirmed.
[0029] TiAlCrON based multi-layers which was based on TiAlN layer but added by Cr and oxigen can improve oxidation proof, but also improve lubrication property without degrading wear resistance and furthermore improves layer adhesion created by lower stress and therefore in high speed dry cutting, superior properties can be obtained. In application field of hot forging and so on, its effects are the same.

Claims

1. Hard layer coated parts, **characterized by** at minimum one or two layers which contain Al, Ti, Cr, N, O.

2. Hard layer coated parts according to claim 1, **characterized by** each layer which consists hard layer has the following chemical analysis:

$$(Al_aTi_bCr_c)\ (N_wO_{1-w}),$$

however, $30 \leqq a \leqq 70,\ 30 \leqq b \leqq 70,\ 0.5 \leqq c \leqq 20,$
$a + b + c = 100,\ 0.7 \leqq w \leqq 0.99,$

3. Hard layer coated parts according to claim 1 or 2, **characterized by** a number of three to thousand layers.

4. Hard layer coated parts according to claims 1 to 3, **characterized by** a layer thickness 5 nm - 2000 nm.

5. Hard layer coated parts according to claims 1 to 3, **characterized by** hard layers with composite of less oxigen-containing A-layer and more oxigen-containing B-layer.

6. Hard layer coated parts according to claim 5, **characterized by** an A-layer with oxigen content of 1 - 10 atomic-% and B-layer with oxigen content of 10 - 30 atomic-%.

7. Hard layer coated parts according to claim 5 or 6, **characterized by** inclination composition of oxigen contents in

A-layer and/or B-layer or entire layers.

8. Hard layer coated parts according to claims 1 to 7, **characterized by** hard layers with a crystal structure of the NaCl type.

9. Hard layer coated parts according to claims 1 to 8, **characterized by** supposing that in X-ray diffraction, the intensity of diffraction of (200)plane is I(200) and the intensity of diffraction of (111) plane is I(111), hard layer coated parts has a feature that I(200)/I(111) is a value of more than 1.

10. Hard layer coated parts according to claims 1 to 9, **characterized by** hard layer crystalization with fine columnar crystals or amorphous.

11. Hard layer coated parts according to claims 1 to 10, **characterized by** a grain diameter of the fine columnar crystals of below 250 nm at a distance of 1000 nm - 1500 nm from the border line between the hard layer and the substrate.

12. Hard layer coated parts according to claims 1 to 11, **characterized by** the compression stress residual in hard layer is less than 3,5 GPa.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 00 10 4982

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	22 August 2000	Ceulemans, J

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	22 August 2000	Ceulemans, J

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A : technological background
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